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THE JOURNAL

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AMERICAN CHEMICAL SOCIETY.

THE RELATION OF TEACHING TO RESEARCH IN CHEMISTRY.[1]

BY W. E. STONE.

IT is eminently proper that this Congress of Chemists should devote some portion of its attention to the teaching of the science. This not alone because it is desirable that chemists be well taught, but also by reason of the prominent place chemistry has secured in the curriculum of every college and university as well as in many high schools. The teaching of chemistry in institutions of learning is a modern innovation, introduced and developed within the memory of living men. The chemical laboratory as a means of instruction was first recognized in America about thirty years ago. Now the chemical lecture room and laboratory form an essential part of the equipment of every institution for higher education. It naturally follows that teachers of chemistry have become numerous, and positions of this kind are the goals toward which many young chemists aspire.

The teaching of chemistry has therefore become a kind of profession with its own peculiar limitations and disappointments as well as its pleasures and aspirations. The discussion of methods and details of teaching chemistry I leave to the speakers who are to follow me. I wish, however, to consider briefly what I regard as a most important feature of didactic chemistry, viz.: *The attitude of teachers of chemistry toward research.*

[1] Opening address by the Chairman of the Section of Didactic Chemistry at the World's Congress of Chemists, Chicago, August 26, 1893.

A survey of the field reveals an interesting comparison between America and Europe in this respect. In the European universities we find the teachers of chemistry including the famous investigators and discoverers. It is a recognized function of the teacher that he be also an investigator. Indeed his appointment to a chair in any of the great universities is dependent upon his standing as an investigator and he retains his position only so long as he maintains this standing. In short one of the necessary qualifications of the teacher is active participation in scientific reseach. Turning to America, two features are at once noticeable. First, a less degree of activity and attainment in scientific research, and second a weaker interest in the spirit of investigation. We must acknowledge that our teaching and the results of our research are still inferior to those attained abroad. The reasons for this are probably complex, but *chiefly* they are to be sought in the relations of our teachers to investigation. Certainly our teachers of chemistry do not lack training, for many of them have been trained under the conditions abroad which we recognize as superior. They do not lack enthusiasm because many are young men with the successes and examples of their European preceptors still in mind. Our American teachers are not at the head in their profession by reason of any lack of training or enthusiasm or material equipments. If I ask then why, any one of my hearers can answer promptly. Every American teacher of chemistry has a common complaint to voice. They will tell you that the demands made upon them as instructors are alone culpable for their meagre contributions to the annals of research. Too many students. Too many hours of teaching. Too many subjects to be taught. These are the counts in the indictment against the conditions under which our American teachers exist.

In short, to sum up the existing *status*. We find ourselves somewhat in the rear of the foremost ranks of investigators and teachers. We find these branches most flourishing where the activity of the investigator is a required qualification for the teacher. In our own country we find these requirements of secondary moment or they are lost sight of entirely. This seems to me not only a great fault in our educational system, but also a very

serious hindrance to the progress of American chemical science.

I am aware that some will fail to see the relation between teaching and research in chemistry and will maintain that the teacher should be only teaching and the investigator only occupied with investigation.

We may regard this matter from the standpoint of the student, from the standpoint of pure science, and finally from the standpoint of the teacher himself.

The student has a right to expect instruction in chemistry either as part of a liberal education or as preparation for a professional career. In either case it is or should be taught, not as a dead and completed science, but as a constantly advancing vital, living science. It is or should be taught as a science of investigation. The only one who can teach it as such must be himself an investigator. No one would maintain that the discoveries of Wöhler or Hoffmann did in any way detract from their effectiveness as teachers. On the contrary, it is apparent that in their characters as investigators they transmitted an inspiration to their pupils which has given to modern chemistry an incalculable impetus.

Again, science looks mainly to teachers for its advancement, since as a rule, they alone have or should have at command the necessary funds, materials, and equipments for the prosecution of researches. Moreover, they alone have or should have the leisure and unbiased mind so essential in the search for truth.

Lastly, the teacher himself has no right to content himself with the single aim of the pedagog. If he would not stagnate he must advance. He must be himself a student standing as interpreter between the unknown and his pupils. The true attitude of the teacher of chemistry toward research is one of interest and active participation in precisely the same degree as he manifests interest in and sympathy with his pupils.

[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE U. S. DEPARTMENT OF AGRICULTURE; SENT BY H. W. WILEY.—No. 7.]

THE ESTIMATION OF SUGARS IN CONDENSED MILK.[1]

BY W. D. BIGELOW AND K. P. MCELROY.

Abstract read before the Washington Chemical Society, February 9, 1893.

IN the course of the work with canned goods, which has been carried on in this laboratory during the past year, it was desired to examine condensed milk.

The brands of condensed milk are commonly divided into two classes,—those which contain cane sugar, and those which do not. When cane sugar is absent, milk sugar may be determined by any of the methods which are used for its estimation in fresh milk. In the analysis of condensed milk which contains cane sugar, it has usually been taken for granted that invert sugar was absent. Dr. Wiley, by whose direction this work was undertaken, desired a method for the estimation of the sugars present in condensed milk, which would take into consideration the possible presence of invert sugar. It was at his instance, therefore, that the work which forms the basis of this paper was undertaken. It is not our intention at this time to review all the methods which have been suggested in the analysis of condensed milk, but merely to record some ideas which have suggested themselves and some methods which have been developed in the progress of this work.

Among the methods which have been suggested for the determination of cane sugar in the presence of milk sugar the following may be cited as being the most important:

(1) In the method which seems to be most frequently employed, the total solids are estimated by evaporating a portion of the liquid and drying it to constant weight; casein, fat, and ash are estimated by the ordinary methods; the milk sugar by reduction with an alkaline copper solution; and the amount of cane sugar is obtained by deducting the milk solids from the total solids. Aside from the fact that a "difference" method is never satisfactory, this method is open to the objection that whatever invert sugar may be present is estimated as milk sugar, and the presence of invert sugar is always possible where

[1] Read before the World's Congress of Chemists, Chicago, August 22, 1893.

cane sugar has been used in preserving condensed milk. It is certainly advisable to employ a method which will detect it when present.

(2) The method suggested by Muter[1] is well worthy of attention. In this method, ten grams[2] of the milk are poured on four grams of hydrated calcium sulphate in a basin and evaporated to perfect dryness with frequent stirring so that nothing sticks to the basin. The dry residue is powdered, macerated with ether, thrown on a dry filter over a tared beaker, and percolated with ether until free from fat. The ether is then evaporated off, and the beaker plus fat weighed, from which the percentage of fat is ascertained. The filter and contents are then transferred to a beaker, twenty cc. of hot (not boiling) water added, and the whole well stirred. Thirty cc. of alcohol (sixty per cent.) are then added, and the mixture is allowed to cool with occasional stirring. When cool it is thrown on a filter placed over a long graduated measure and washed with proof spirits (two volumes of water to one of sixty per cent. alcohol) until the filtrate measures 120 cc., at which point the extraction is usually complete. The filtrate is then divided into two equal parts and one portion evaporated in a platinum dish, and then placed in a drying oven at 100° till constant weight is obtained. The weight is then noted, the dish ignited for some time at a dull red heat and again weighed, when the total weight less dish plus ash gives the total sugar, which is multiplied by twenty to get percentage. Evaporate the alcohol from the other portion of the filtrate, wash into a beaker, and determine lactose by means of Fehling's solution. The weight of cuprous oxide thus obtained is multiplied by twenty and calculated from 146 to 100. The amount of milk sugar thus found is deducted from the total sugar, and the result, if over 0.5, is put down as cane sugar. If over 0.5 and under 1.0, a rebate of 0.2 per cent., and if over 1.0 and under 1.5, an allowance of 0.1 per cent., is to be made. But if the amount reaches over 2.0 per

[1] *Analyst*, *1880*, 5, 37.

[2] This method is proposed for the examination of fresh milk which has been adulterated with condensed milk. If it is desired to apply it to condensed milk, two to three grams should be taken instead of ten grams, and the corresponding correction made in the method of calculation.

cent. it may be taken as it stands. This method is somewhat tedious, and in general is open to the same objections as the first one cited. It is also found that when a large amount of cane sugar is present considerable difficulty is experienced in the extraction of both sugar and fat.

(3) The inversion of the cane sugar by means of mineral acids: A number of methods have been suggested which may be classed under this head. They are all open to the objection that milk sugar also undergoes hydrolysis when subjected to the action of mineral acids. It has been suggested that a blank containing the same amount of milk sugar as the milk under examination be run with each experiment. This is not practicable, however, as the action of milk sugar under these circumstances is far from uniform.

(4) Shenstone[1] determines both sugars by the aid of the polariscope, milk sugar by copper reduction, and estimates the cane sugar by difference. His method, briefly stated, is as follows: Dilute thirty grams of condensed milk, boil to obtain a normal rotation of the milk sugar, cool, make up to ninety-seven cc. with water, and add three cc. of acid mercurous nitrate. The solution is then mixed by pouring between two beakers, and filtered. One portion of the filtrate is then polarized; this gives the combined readings of sucrose and lactose. Ten cc. of the filtrate are then diluted to 100 cc., and the milk sugar is determined by either Fehling's or Pavy's solution; the combined reading obtained from both sugars less that representing the amount of milk sugar present being equal to the polarization of the cane sugar present. This method has also the disadvantage that it does not indicate the presence of invert sugar. Another objection which may be urged is that inversion is likely to be caused by the nitric acid present in the solution. The author states that if the sugars be determined as soon as possible after the clarification of the milk no inversion can be detected. It is evident, however, that should even a slight inversion occur, a decided error in the amount of sucrose obtained would result. In the first place, the invert sugar would reduce sufficient cuprous oxide to indicate almost

[1] *Analyst*, *1888*, **13**, 222.

twice its weight of milk sugar. Then, of course, the polarization of the combined sugars would be reduced. Thus, the sucrose would be obtained by subtracting a figure too large for the amount of milk sugar from one too small for the combined polarization of the two sugars.

(5) Stokes and Bodmer[1] have suggested that the cane sugar be determined by inversion with citric acid. The details of their method are as follows: Dilute the milk and place in a burette. Hang on to the lower end of the burette a flask (100 cc.) by means of a rubber tube fitted with a screw clip. The flask should be provided with another tube for the exit of ammonia vapors. Place in the flask forty cc. of Pavy's solution, boil, and gradually add the sugar solution till the blue color disappears. Read the burette. Into another flask containing forty cc. of Pavy's solution run the amount of sugar solution (less two-tenths) found necessary to decolorize in the first experiment, and boil. Should the liquid remain blue, it shows that the first reading was correct within 0.1 cc. Next boil another portion of the diluted milk with citric acid in sufficient amount to form two per cent. of the solution, for ten minutes, cool, neutralize with ammonia and repeat the titration. Cane sugar is completely inverted by boiling with two per cent. of citric acid, while milk sugar is not affected. The reducing power of milk sugar may be taken as being fifty-two per cent. of that of glucose, using Pavy's solution. The dilution of the milk should be such that from six to twelve cc. are required for the titration. This method does not seem to have come into general use, although it has been tried by several and is said to give good results. The invert sugar is not taken into account. Shenstone[2] proposes a modification of the apparatus used by Stokes and Bodmer by which he replaces the ammonia driven off during the operation.

The Determination of Cane Sugar.—It is well known that in the inversion of cane sugar by means of acids, the results are greatly influenced by the apparatus used and the details of the operation. For this reason it was thought best to examine the

[1] *Analyst*, *1885*, 10, 62.

[2] *Analyst*, *1888*, 13, 222.

methods for the inversion of cane sugar by means of invertase.[1] This method was first proposed by Kjeldahl[2] for the estimation of the sucrose in barley and malt. He found that results obtained by its use were more accurate than those obtained by inversion with acids. Later it was used by O'Sullivan[3] for the estimation of sucrose in cereals. In 1890, O'Sullivan and Tompson[4] made a careful study of this method and published an account of their work together with a bibliography of invertase and directions for its preparation.

Instead of preparing the invertase itself as they here directed, we followed the method suggested by them in a later paper[5] and used the yeast liquor obtained by the decomposition of brewers' yeast.

For the preparation of this liquid, brewers' yeast was allowed to stand one month at the temperature of 15°. The product, which was then mostly liquid, was filtered, and for the purpose of preservation alcohol was added till the solution contained ten to twelve per cent. absolute alcohol. The mixture was then allowed to stand for a few days and filtered.

The first series of experiments was made to determine the amount of yeast liquor required to obtain complete inversion. The amount of yeast liquor used varied from one to three cc. and the time was four hours.

About 200 grams of granulated sugar were dissolved in one liter of water. For each inversion fifty cc. of this solution were transferred to a hundred cc. flask. Two of these portions were made up to the 100 cc. mark, and the solutions polarized without inversion to obtain the direct reading. The solution polarized 38.3° on the Schmidt and Haensch half-shadow instrument in a 200 mm. tube. The flasks which contained sugar intended for inversion were placed in a large water bath, the temperature of which was held at 55° throughout the operation. As

[1] Richmond and Boseley (*Analyst, July, 1893*) suggest that this method would probably be suitable for the determination of cane sugar in condensed milk. Our own work, however, was done before that time.

[2] *Meddelelser, 1881*, 337.

[3] *J. Chem. Soc., Trans., 1886*, **49**, 58.

[4] *J. Chem. Soc., Trans., 1890*, **57**, 834.

[5] *J. Chem. Soc., Trans., 1891*, **59**, 46.

soon as the proper temperature was reached the yeast liquor was added. In order to hold the flasks in position in the bath, a frame was constructed of parallel strips placed about one inch apart and small nails were driven at intervals of about two inches along the top of the strips. The necks of the flasks were then placed between these strips and held in position by means of rubber bands which were stretched across between the nails. At the end of four hours the operation was stopped. A little corrosive sublimate was added to the contents of each flask to prevent any fermentation which might be caused by the yeast as the solution cooled. Some alumina cream was also added to assist in the clarification. The solutions were then cooled to about the temperature of the room, made up to the 100 cc. mark, thoroughly mixed by shaking, filtered, and polarized. The result of this experiment is given in table 1.

TABLE 1.

Yeast liquor, cc.	Total liquid in cc.	Cane sugar added, grams.	Cane sugar found, grams.
1	51	9.976	9.690
1	51	9.976	9.664
2	52	9.976	9.846
2	52	9.976	9.820
3	53	9.976	9.898
3	53	9.976	9.768
3	53	9.976	9.716

On examining this table, it will be evident that the amount of cane sugar indicated is too low in every case.

Another series of inversions was then started in which the sucrose solution was of the same strength as in the previous series, though only half as much (about five grams) was taken for each inversion. The direct polarization of one of these portions was found to be 19.15° on the sugar scale. The time allowed was five hours and the amount of yeast liquor used was five cc. The results of this series will be found in table 2, which is given below:

TABLE 2.

Yeast liquor, cc.	Total liquid in cc.	Cane sugar added, grams.	Cane sugar found, grams.
5	30	4.988	5.001
5	30	4.988	4.975
5	30	4.988	4.975
5	30	4.988	5.001
5	30	4.988	4.988
7	32	4.988	5.001

It was also suggested by O'Sullivan and Tompson[1] that brewers' yeast be used instead of invertase or yeast liquor. They directed that fresh brewers' yeast be added to the solution to be inverted to the amount of about one-tenth the weight of the sucrose present. On account of the greater uniformity of ordinary compressed yeast, however, and the ease with which it can always be obtained, a series of inversions was made with it and its action compared with that of the yeast liquor used in the preceding experiments. For this purpose the cane sugar solution was prepared as before, and some portions containing ten, and others containing five grams, were taken for the inversion. The time allowed for this series was four hours. The results obtained by this series of inversions are given in table 3.

TABLE 3.

Yeast, grams.	Total liquid in cc.	Cane sugar added, grams.	Cane sugar found, grams.
1	60	9.976	9.989
1	60	9.976	9.872
1	60	9.976	9.872
2	70	9.976	9.846
2	70	....	....
2	70	....	....
2	45	4.988	4.845
2	45	4.988	4.897

These results are too low. In order to determine whether this is due to the fact that not enough time was allowed for complete inversion, or an insufficient amount of yeast was employed, two more sets of inversions were begun, in one of which (table 4), five grams, in the other (table 5), ten grams, of cane sugar were employed.

TABLE 4.

Yeast, grams.	Total liquid in cc.	Cane sugar added, grams.	Cane sugar found, grams.
1	25	4.988	4.975
1	25	4.988	4.988
1	25	4.988	4.949
2	25	4.988	4.975
2	25	4.988	4.949
2	25	4.988	5.001

[1] *J. Chem. Soc., Trans.*, *1891*, 59, 46.

TABLE 5.

Yeast, grams.	Total liquid in cc.	Cane sugar added, grams.	Cane sugar found, grams.
2	50	9.976	9.950
2	50	9.976	9.950
5	50	9.976	9.976
5	50	9.976	10.002
5	50	9.976	9.976
5	50	9.976	9.976

In each case the inversion was allowed to proceed for five hours. Several of the inversions were allowed to proceed for seven hours, and in no case was any destructive action upon the invert sugar evident.

From these results we infer:

(1) That the results obtained by means of compressed yeast compare very favorably with those obtained by the use of yeast liquor, and since this is more readily obtained and is more convenient it is to be preferred.

(2) That the time necessary for complete inversion is five hours.

(3) That while fair results may be obtained with an amount of yeast equal to one-fifth the weight of the sucrose present, the best results are to be obtained with an amount equal to one-half the weight of the sucrose it is desired to invert. It will be remembered that O'Sullivan and Tompson[1] added brewers' yeast to the amount of one-tenth the weight of sucrose which it was desired to invert. This is due to the fact that the per cent. of invertase contained in compressed yeast is less than in brewers' yeast.

The next step was to determine the effect of invertase upon a pure solution of milk sugar. There seems to be no literature upon this subject. Dastre[2] makes the statement that lactose is inverted by the invertive ferment present in yeast. He seems, however, to base his opinion on the fact that lactose disappears in the presence of yeast, and cites no experiment which would seem to justify the statement that this sugar is hydrolyzed.

[1] *J. Chem. Soc., Trans.*, *1891*, 59, 46.

[2] *Compt. Rend.*, *1883*, 96, 932.

Milk sugar was dissolved in water, the solution boiled to obtain the normal rotation of milk sugar, cooled to room temperature, and portions of fifty cc. each measured into 100 cc. flasks. Two of these were diluted to the mark and polarized. The reading was 5.2°. The others were subjected to the action of invertase for five hours at 55°, some with yeast liquor, others with compressed yeast. The polarization of the solution before and after the operation is given in tables 6 and 7.

TABLE 6.

Yeast liquor, cc.	Total liquid in cc.	Original polarization.	Polarization after inversion.
1	51	5.2	5.3
1	51	5.2	5.1
2	52	5.2	5.1
2	52	5.2	5.2
2	52	5.2	5.2
3	53	5.2	5.3
3	53	5.2	5.2

TABLE 7.

Yeast, grams.	Total liquid in cc.	Original polarization.	Polarization after inversion.
2	50	5.2	5.1
2	50	5.2	5.3
2	50	5.2	5.3
5	50	5.2	5.2
5	50	5.2	5.0

From these figures we feel justified in saying that the inversion noted by Dastre must have been due to some other cause than the influence of the invertase present in yeast.

The fact being thus established that milk sugar is not affected by the action of invertase, it was next undertaken to invert cane sugar in the presence of milk sugar by this method.

Solutions of the two sugars were prepared of about the same strength as was used in the preceding experiments. About 600 grams of granulated sugar were dissolved in three liters of water. Approximately 100 grams of milk sugar were dissolved in three liters of water. This solution was boiled for some time, and then cooled to room temperature. Fifty cc. of the cane sugar solution diluted to 100 cc. and polarized gave a reading of 38.2° on the sugar scale.

In this set of inversions, as in the previous ones, 100 cc.

flasks were used. Into each flask were placed fifty cc. of the sucrose solution and twenty-five cc. of the lactose solution, making about twelve parts of the former to one of the latter. The operation was conducted just as in the inversions of the cane sugar alone, and was allowed to proceed for five hours. The results are given in table 8.

TABLE 8.

Yeast liquor, cc.	Milk sugar, grams.	Total liquid in cc.	Cane sugar added, grams.	Cane sugar found, grams.
1	0.86	76	9.950	9.794
2	0.86	77	9.950	9.898
2	0.86	77	9.950	9.924
3	0.86	77	9.950	9.950
3	0.86	78	9.950	9.924
3	0.86	78	9.950	9.950

In this experiment the amount of yeast liquor used varied from one to three cc. for each inversion. It will be seen from the table that the inversion was not complete in those samples which contained less than three cc. of yeast liquor.

It was then attempted to invert cane sugar mixed with about one-third its weight of milk sugar. For that purpose twenty-five cc. of a cane sugar solution, fifty cc. of a milk sugar solution, of the same strength as was used in the preceding experiments, were transferred to each flask used in this series of inversions. Twenty-five cc. of the cane sugar diluted to 100 cc. and polarized gave a reading of 19.1° and contained about five grams of sucrose. After five hours the inversion was interrupted. The results are given in table 9.

TABLE 9.

Yeast liquor, cc.	Total liquid in cc.	Milk sugar added, grams.	Cane sugar added, grams.	Cane sugar found, grams.
1	76	1.716	4.975	4.923
1	76	1.716	4.975	4.975
1	76	1.716	4.975	4.975
2	77	1.716	4.975	4.923
2	77	1.716	4.975	4.949
2	77	1.716	4.975	4.975
3	78	1.716	4.975	4.975
3	78	1.716	4.975	4.949
3	78	1.716	4.975	4.975
3	78	1.716	4.975	4.949

It was next desired to study the influence of compressed yeast on solutions containing both sucrose and lactose in the proportion of twelve of the former to one of the latter. Each portion contained about ten grams of cane sugar. The inversion was interrupted after four hours. In table 10 are given the results of the inversion.

TABLE 10.

Yeast, grams.	Total liquid in cc.	Milk sugar added, grams.	Cane sugar added, grams.	Cane sugar found, grams.
1	85	0.858	9.950	9.820
1	85	0.858	9.950	9.898
1	85	0.858	9.950	9.924
1	85	0.858	9.950	9.820
1	85	0.858	9.950	9.898
2	95	0.858	9.950	9.872
2	95	0.858	9.950	9.898

These results are rather low, probably on account of the fact that sufficient time was not allowed for the complete inversion of this amount of sucrose.

The same amount of cane sugar was then inverted in the presence of twice the amount of milk sugar. The time allowed was in some cases four (table 11), in some, five hours (table 12).

TABLE 11.

Yeast, grams.	Total liquid in cc.	Milk sugar added, grams.	Cane sugar added, grams.	Cane sugar found, grams.
1	100	1.716	9.950	9.898
1	100	1.716	6.950	9.846
1	100	1.716	9.950	9.950
2	100	1.716	9.950	9.794
2	100	1.716	9.950	9.846
2	100	1.716	9.950	9.846

TABLE 12.

Yeast, grams.	Total liquid in cc.	Milk sugar added, grams.	Cane sugar added, grams.	Cane sugar found, grams.
2	100	1.716	9.950	9.898
2	100	1.716	9.950	9.976
2	100	1.716	9.950	9.950
5	100	1.716	9.950	9.924
5	100	1.716	9.950	9.950

The examination of these tables confirms the former experi-

ments with regard to the time necessary for complete fermentation.

In this case, as in all others in which the volume of the liquid was too great to make use of a 100 cc. flask, a 200 cc. flask was used instead, and the reading obtained was multiplied by two.

A mixture of cane and milk sugar was then made, in which the sugars were present in about the same proportion as they are usually found in condensed milk, *i. e.*, three parts of cane sugar to one of milk sugar. Approximately five grams of sucrose were present. The time allowed for this set of inversions was five hours, and the result, as will be seen in table 13, was entirely satisfactory.

TABLE 13.

Yeast, grams.	Total liquid in cc.	Milk sugar added, grams.	Cane sugar added, grams.	Cane sugar found, grams.
1	75	1.716	4.975	4.983
1	75	1.716	4.975	4.949
1	75	1.716	4.975	4.975
1	75	1.716	4.975	4.975
1	75	1.716	4.975	4.923
2	75	1.716	4.975	5.001
2	75	1.716	4.975	4.949
2	75	1.716	4.975	4.975

A sample of whole milk was then taken, fifty cc. introduced into a 100 cc. flask, three cc. mercuric iodide solution[1] and a little alumina cream added, the solution made up to the mark, filtered, and polarized. Similar portions of the milk were heated to 80° to destroy any ferments which might be present, allowed to cool to 55° and treated as before; some with yeast liquor and others with compressed yeast. Some of these were kept at this temperature for four hours, and others for five hours. Then they were clarified as before, cooled, made up to the mark, filtered, and polarized. On examining table 14, it will be seen that, allowing for the usual error of polarization, the milk sugar had not been in any way affected by the action of the invertase. A portion of this milk was diluted to some extent before the portions for inversion were measured out. These, as will be seen by the table, read 6.1° on the sugar scale before inversion.

[1] Potassium iodide 33.2 grams, mercuric chloride 13.5 grams, acetic acid 20 cc., water 64 cc.

TABLE 14.

Yeast liquor, cc.	Total liquid in cc.	Milk, cc.	Polarization before inversion.	Polarization after inversion.	Time in hours.
3	53	50	8.2	8.1	4
3	53	50	8.2	8.2	4
3	53	50	8.2	8.3	4
3	53	50	8.2	8.2	4
3	53	50	8.2	8.2	4
3	53	50	8.2	8.2	4
3	53	50	8.2	8.1	4
3	53	50	8.2	8.2	4
3	53	50	8.2	8.3	4
3	53	50	8.2	8.3	4
3	53	50	8.2	8.2	5
3	53	50	8.2	8.2	5
3	53	50	8.2	8.2	5
3	53	50	8.2	8.1	5
3	53	50	6.1	6.1	5
3	53	50	6.1	6.0	5
5	55	50	6.1	5.9	5
5	55	50	6.1	6.1	5
5	55	50	6.1	6.1	5
5	55	50	6.1	6.2	5

The influence of compressed yeast upon whole milk was next tried. This series is a repetition of the previous one, with the substitution of compressed yeast for yeast liquor. The results are given in table 15.

TABLE 15.

Yeast, grams.	Total liquid in cc.	Milk, cc.	Polarization before inversion.	Polarization after inversion.	Time in hours.
2	70	50	8.2	8.0	4
2	70	50	8.2	8.2	4
2	70	50	8.2	8.2	4
2	70	50	8.2	8.3	4
2	70	50	8.2	8.0	4
2	70	50	8.2	8.2	4
2	70	50	8.2	8.2	4
2	70	50	8.2	8.2	4
2	70	50	8.2	8.1	4
2	70	50	8.2	8.2	5
2	70	50	8.2	8.3	5
2	70	50	6.1	6.0	5
2	70	50	6.1	6.1	5
2	70	50	6.1	6.1	5

From these results it appears that if there is any inversion of milk sugar produced by the action of invertase, the amount must be so small that it can not be detected by the polariscope.

The next step taken was to mix a solution of cane sugar with whole milk, and invert the sucrose in this mixture. For this purpose approximately 400 grams of granulated sugar were dissolved in one liter of water. Twenty-five cc. of this solution diluted to 100 cc. gave a reading of 39.0° on the sugar scale. Fifty cc. of the milk which was to be used in this experiment were placed in a 100 cc. flask, clarified as given above by means of mercuric iodide dissolved in acetic acid, and alumina cream, made up to 100 cc., filtered, and polarized. The reading was 8.2°. To another fifty cc. of the same milk twenty-five cc. of the cane sugar solution were added, clarified as before, made up to the mark, filtered, and polarized. The filtrate read 48.2°. From these figures the volume of the solids precipitated from fifty cc. of the milk was estimated to be 2.5 cc. For the purpose of inverting cane sugar in solution in milk, portions of fifty cc. each of the milk were measured into 100 cc. flasks, twenty-five cc. of the solution of cane sugar were added to each portion, and the mixture treated with yeast liquor in the same manner as in the inversion of sucrose in aqueous solution. As soon as the contents of the flasks had reached the temperature of 55°, the yeast liquor was added. This temperature was held constant for four hours. Three cc. of the solution of mercuric iodide in acetic acid and a little alumina cream were then added, the flasks shaken gently, cooled, made up to the mark, filtered, and the filtrate polarized. The results obtained by this method are given in table 16.

TABLE 16.

Yeast liquor, cc.	Total liquid in cc.	Milk, cc.	Cane sugar added, grams.	Cane sugar found, grams.
3	80	50	10.158	10.054
3	80	50	10.158	10.080
3	80	50	10.158	10.132
3	80	50	10.158	10.132
3	80	50	10.158	9.976

A solution of cane sugar was also made which contained half the amount of sucrose given above. Similar mixtures inverted

as in the preceding experiment for four hours showed that the sucrose was completely inverted, as may be seen in table 17.

TABLE 17.

Yeast liquor, cc.	Total liquid in cc.	Milk, cc.	Cane sugar added, grams.	Cane sugar found, grams.
3	80	50	5.079	5.079
3	80	50	5.079	5.079
5	80	50	5.079	5.105

A comparison of tables 16 and 17 shows that better results were obtained with five grams of sucrose than with ten grams. It was thought that a five hour inversion would give better results in the presence of ten grams of sucrose. Twenty-five cc. of an aqueous solution containing that amount of cane sugar were added to fifty cc. of milk, and the mixture subjected to the action of yeast liquor for five hours. The results as given in table 18 would seem to indicate that five hours are necessary for complete inversion.

TABLE 18.

Yeast liquor, cc.	Total liquid in cc.	Milk, cc.	Cane sugar added, grams.	Cane sugar found, grams.
3	80	50	10.158	10.162
3	80	50	10.158	10.158
3	80	50	10.158	10.210
3	80	50	10.158	10.132
3	80	50	10.158	10.162
3	80	50	10.158	10.106
3	80	50	10.158	10.210
3	80	50	10.158	10.210
3	80	50	10.158	10.236
4	80	50	10.158	10.158
4	80	50	10.158	10.184
4	80	50	10.158	10.158
4	80	50	10.158	10.158
4	80	50	10.158	10.080
4	80	50	10.158	10.236

It was next desired to study the influence of compressed yeast on a solution of sucrose in milk. For this purpose about 180 grams of granulated sugar were dissolved in one liter of water and the solution polarized. Twenty-five cc. of this solution were mixed with fifty cc. of milk for each inversion. As in all former cases compressed yeast gave the same result as the

liquor prepared from brewers' yeast had done. The results are given in table 19.

TABLE 19.

Yeast, grams.	Total liquid in cc.	Milk, cc.	Cane sugar added, grams.	Cane sugar found, grams.
2	75	50	4.611	4.637
2	75	50	4.611	4.585
2	75	50	4.611	4.637
2	75	50	4.611	4.611
2	75	50	4.611	4.585
2	75	50	4.611	4.611

The time allowed for this operation was but four hours, but the amount of sugar in each flask was less than five grams and the inversion was complete.

It was next undertaken to invert ten grams of cane sugar by means of yeast. This amount of granulated sugar dissolved in twenty-five cc. of water was added to fifty cc. of milk, the mixture clarified, made up to the mark, filtered, and the filtrate polarized as before. The reading was 48.2°. Fifty cc. of the milk treated in the same way gave a reading of 8.2°. The reading of the sucrose added, therefore, was taken as 40.0°. At the end of four hours the operation was interrupted. The sucrose found is given in table 20.

TABLE 20.

Yeast, grams.	Total liquid in cc.	Milk, cc.	Cane sugar added, grams.	Cane sugar found, grams.
2	75	50	10.158	10.132
2	75	50	10.158	10.080
2	75	50	10.158	10.210
2	75	50	10.158	10.158
2	75	50	10.158	10.132
2	75	50	10.158	10.206
2	75	50	10.158	10.132

The time in this case was not sufficient to permit complete inversion.

Other portions of the solutions used in the preceding set of inversions were treated in the same manner, except that the inversion was allowed to proceed for five hours. The results are given in table 21.

TABLE 21.

Yeast, grams.	Total liquid in cc.	Milk, cc.	Cane sugar added, grams.	Cane sugar found, grams.
2	75	50	10.158	10.184
2	75	50	10.158	10.184
2	75	50	10.158	10.184
2	75	50	10.158	10.210
2	75	50	10.158	10.210
2	75	50	10.158	10.158
2	75	50	10.158	10.028
2	75	50	10.158	10.158
2	75	50	10.158	10.210

The comparison of tables 19, 20, and 21, confirms the conclusions drawn from the results of the corresponding work with the liquor from brewers' yeast.

From these experiments it is evident that this method may be applied to the determination of sucrose in the presence of milk with results which are entirely satisfactory.

Four determinations of sucrose in one of our most popular brands of condensed milk gave the following results:

40.0 per cent. 40.0 per cent. 39.9 per cent. 40.2 per cent.

After this article was prepared Richmond and Boseley[1] made the statement that the polariscope could not be used for the examination of condensed milk, because of the effect of the heat necessary to condense the milk on the optical properties of milk sugar. Thus far we have met with no difficulty from this source.

Milk Sugar.—The method for the determination of milk sugar which is suggested below is intended to be used only in the presence of invert sugar. It is evident that when this is present the milk sugar can be determined neither by copper reduction nor by polarization.

Under these circumstances the most convenient solution of the problem seemed to be the destruction of the cane and invert sugars by fermentation, and the subsequent determination of the lactose by means of the polariscope, or by the copper reduction method. It is, however, well known that the complete fermentation of sucrose is a difficult thing to accomplish, the trouble being largely due to the lactic ferment which invariably occurs in saccharine liquids fermenting under ordinary circumstances.

[1] *Analyst*, *1893*, **18**, 141.

It is equally well known that lactose disappears in the presence of common yeast. This is also probably due to the lactic ferment. Certain inorganic acids and salts, especially hydrofluoric acid and fluorides, have the power of preventing the lactic acid fermentation, and of accelerating, retarding, or even entirely stopping the yeast fermentation, according to the amount employed.

Although fluorides have been extensively used for this purpose in breweries and distilleries, no attempt has yet been made to apply their action to an analytical method.

It seemed desirable then to investigate this subject in order to determine whether their action was sufficiently complete to form the basis of an analytical method. Our first experiments were made to determine whether cane sugar could be completely fermented by yeast in the presence of fluorides. About 1,200 grams of granulated sugar were dissolved in water and the solution made up to six liters. One hundred and seventeen portions of this solution of fifty cc. each (each containing about ten grams of sucrose) were placed in flasks with one-half cake Fleischman's compressed yeast, together with amounts of potassium fluoride varying from five to thirty-five mgms. to 100 cc. of the solution.

These flasks were divided into three lots which were designated as lots A, B, and C. Lot A was kept at a temperature of from 11° to 15°, lot B from 20° to 25°, and lot C in a room the temperature of which varied from 15° to 35°.

In each of these lots the flasks were arranged in thirteen sets of three each. The flasks of each set all contained the same amount of fluoride, and a blank containing no sugar was run with each set. One flask from each set was used for qualitative tests with Fehling's solution. The others were polarized.

After standing three days all samples showed heavy copper reduction. After five days all samples of lot A showed heavy copper reduction, while those samples of lots B and C, which contained the lower amounts of fluorides, showed but light copper reduction. Lot C had progressed farther than lot B.

After seven days the fermentation in lots B and C was almost complete. The fermentation in lot B seemed to be the more

favorable. After eight days the Fehling's solution gave but a slight reaction in lot B and but slightly heavier in lot C. On the tenth day lots B and C were polarized and the sugar was found to be entirely destroyed. Lot A required a much longer time for complete fermentation than lots B and C, sugar still being present after three weeks' standing. After twenty-five days, however, the fermentation was complete. From these experiments we conclude:

(1) That it is possible and entirely practicable to completely ferment cane sugar by means of yeast in the presence of potassium fluoride.

(2) That with from fifteen to thirty mgms. of fluoride per 100 cc. of the solution the fermentation is most satisfactory.

(3) That the best temperature for complete fermentation is from 20° to 25°; and

(4) That under these circumstances eight days is sufficient for complete fermentation.

The next step was to determine the action of yeast on milk sugar in the presence of fluorides. Approximately 160 grams of milk sugar were dissolved in one liter of water and the solution boiled for some minutes. One hundred cc. of this solution placed in a 200 cc. flask, filled up to the mark and polarized, gave a reading of 24.4° on the sugar scale. Similar portions of the solution were treated with yeast and fluoride as shown in the table and set aside for a number of days. In this series, however, there were six determinations in each set instead of three as in the preceding set. Beginning with the eighth day these solutions were polarized at intervals of from two to three days. In table 22 are given all the readings which were taken in this series.

TABLE 22.

Lactose, grams.	Mgms. KF, per 100 cc.	Total liquid in cc.	Original polarization.	Polarization after 8 days.	Polarization after 11 days.	Polarization after 13 days.	Polarization after 16 days.	Polarization after 18 days.	Polarization after 35 days.
8	..	100	24.4	23.4	22.8	21.6	20.4	....	....
8	15	103	24.4	24.4	24.4	24.0	23.6	....	....
8	20	105	24.4	24.4	24.4	24.2	24.0	....	....
8	30	108	24.4	24.4	24.4	24.3	23.8	....	20.0
8	45	110	24.4	24.4	24.4	24.3	24.0	....	18.6
8	60	112	24.4	24.6	24.4	24.2	24.3	24.2	19.2

As before said, it is usually stated that milk sugar is slowly consumed by common yeast. Our own experiments without the use of fluorides confirm this idea, as is shown in table 22. It will be seen that on the eleventh day the sample which contained no fluoride read 1.6° lower than those which contained fluoride. It was also impossible to obtain a good clarification on that day, probably on account of the lactic acid ferment which was growing in numbers and which passes through the filter and renders the filtrate turbid. On the sixteenth day the reading was made only with extreme difficulty, while on the eighteenth day it was impossible to effect a clarification which would enable us to read the solution at all.

The next step was the fermentation of mixtures of cane and milk sugar. Separate solutions of both sugars were made in the same manner as in the two preceding series of experiments. This series was treated just as the previous one except that six grams of sucrose were introduced into each flask with the eight grams of lactose. At the temperature at which these experiments were conducted (18° to 20°) thirteen days were required for the complete fermentation of the sucrose. It is believed that ten days would have been sufficient if the temperature had been kept at about 25°. The milk sugar solution used polarized 24.3°. All the readings taken in this series are given in table 23.

TABLE 23.

Mgms. KF. per 100 cc.	Total liquid in cc.	Polarization after 5 days.	Polarization after 8 days.	Polarization after 11 days.	Polarization after 13 days.	Polarization after 16 days.	Polarization after 18 days.	Polarization after 35 days.
..	150	23.1	24.0	23.8	23.8	24.0	....	....
15	154	24.1	23.7	24.0	24.0	24.1	24.0	23.4
20	156	23.4	23.1	24.0	24.3	24.3	24.3	23.1
30	158	23.4	23.4	23.7	24.3	24.3	24.3	23.1
40	162	23.1	23.1	24.0	24.3	24.4	24.3	23.7
55	166	23.1	23.7	24.0	23.8	24.0	24.3	24.3

On examining this table it will be seen that the sample which contained no fluoride, and the one which contained but fifteen mgms., did not reach the polarization of the lactose, for before the fermentation of the sucrose was complete the lactic acid fermentation set in to such an extent as to retard the action of the yeast. On the eleventh day the filtrate from the solution con-

taining no fluoride was slightly turbid; on the thirteenth day the reading could be taken only with difficulty, while on the sixteenth day only an approximate reading could be obtained. On the thirteenth day the filtrate from the solution containing fifteen mgms. of fluoride was slightly turbid, and this turbidity increased with each successive polarization of samples containing this amount of fluoride.

A mixture of ten grams of cane sugar and eight grams of milk sugar was next subjected to the action of yeast in the presence of potassium fluoride. The original polarization of the milk sugar was 24.4°. The temperature was held at from 25° to 30° throughout. The results are given in table 24.

TABLE 24.

Mgms. KF, per 100 cc.	Total liquid in cc.	Polarization after 8 days.	Polarization after 11 days.	Polarization after 13 days.
15	113	24.0	24.2	24.4
25	115	24.3	24.4	24.3
50	120	24.5	24.3	24.4

It is thus evident that the presence of milk sugar does not retard the fermentation of sucrose.

It was next undertaken to ascertain whether anything is present in milk which would interfere with the action of the fluoride. For this purpose a sample of whole milk was taken and divided into portions of fifty cc. each, to which varying amounts of fluoride were added. To each of these portions one-half cake of compressed yeast was added. The polariscopic reading of the milk before fermentation was 14.4° on the sugar scale. The readings which were obtained on five, eight, and eleven days after the beginning of the operation are given in table 25.

TABLE 25.

Mgms. KF, per 100 cc.	Original polarization.	Polarization after 5 days.	Polarization after 8 days.	Polarization after 11 days.
0	14.4	..	4.8	2.2
0	13.8	8.0	5.4	1.4
6	14.4	9.6	3.8	3.2
6	14.4	10.2	..	4.8
11	14.4	11.6	6.0	..
17	14.4	10.4	5.2	3.2
40	13.8	10.0	8.8	2.4

It will be seen that in every case there was a decided decrease in the polarized reading of the solution the fifth day and that this became more apparent the longer the operation was allowed to continue. There is evidently something present in milk which did not occur in the aqueous solution of the pure sugars which prevented in a measure the action of the fluoride in preserving lactose. It will be observed that the destruction of the lactose was less in those solutions which contained fluorides than in those which did not, and that for the first eight days the lactose was better preserved in those solutions which contained the larger amounts of fluorides than in those which contained the smaller amounts.

It was at first thought that there might be germs present in the milk, which would account to some extent for the results obtained in the experiment just cited. For this reason a sample of milk was heated to 80° and kept at that temperature for one hour, raising the temperature at the last to the boiling point for a few minutes. The sample was then allowed to cool and after four hours it was heated again as before. It was then cooled, portions of fifty cc. each were measured out and subjected to the action of yeast in the presence of fluoride as before. The original reading of the milk was 14.4°. After nine days the reading was 50.8°. This indicates that the destruction of milk sugar noted was not due to any ferments present in the milk.

It was then thought that this might be due to the presence of the casein in the milk. To ascertain this, one liter of milk was heated for about thirty minutes with ten cc. glacial acetic acid, filtered, and the filtrate polarized. Portions of this filtrate of 100 cc. each were then introduced into flasks and treated with yeast, and with amounts of potassium fluoride varying from 30 to 120 mgms. per 100 cc. of the solution. In this experiment different degrees of dilution in the filtrate were employed. The polarization of a portion of the clarified milk used in each case is given in table 26, together with the amount of fluoride used and the polarization after a number of days.

TABLE 26.

Mgms. KF, per 100 cc.	Original polarization.	Polarization after 8 days.	Polarization after 18 days.	Polarization after 20 days.
30	13.6	6.4	..	..
50	13.6	6.4	..	..
60	13.6	7.0	..	..
70	10.8	5.6	2.0	0
80	10.8	6.2	1.8	0
100	9.4	7.2	3.6	..
120	9.4	4.8	..	..

It is evident from this table that the amount of milk sugar destroyed was not by any means proportional to the amount of fluorides employed. The milk sugar seems to have been steadily consumed until after twenty days lactose could not be detected by means of the polariscope, although in this case the filtrate was so turbid that a slight error may have been made in the reading.

A sample of milk was then clarified by heating to 80° for one-half hour with a 100 cc. of a normal solution of oxalic acid to each liter. The mixture was then filtered, potassium hydroxide added to the filtrate until the reaction was almost neutral, and again filtered. This filtrate was then subjected to the action of yeast and potassium fluoride with the results given in table 27.

TABLE 27.

Mgms. KF, per 100 cc.	Original polarization.	Polarization after 5 days.	Polarization after 8 days.	Polarization after 14 days.
20	9.9	7.0	..	..
30	9.9	7.8	..	6.2
40	9.9	..	..	5.2
60	9.9	7.8	..	..
0	7.4	5.7	4.9	2.7
40	7.4	6.2	5.4	3.8

When it was desired to polarize samples of this set, they were first clarified by means of the addition of a solution of mercuric iodide in acetic acid, and a little alumina cream, made up to the mark, thoroughly mixed, and filtered. On the addition of the mercury solution a strong evolution of carbon dioxide was observed. It is probable that the potassium oxalate resulting from the neutralization of the oxalic acid was converted into potassium carbonate by the action of the yeast. This, of course, would

soon make the solution alkaline and thus interfere with the action of the fluoride.

A sample of milk was then clarified with glacial phosphoric acid in the same manner as in the preceding experiment, filtered, neutralized, and filtered again. The results as given in table 28 confirm those of the preceding experiment.

TABLE 28.

Mgms. KF. per 100 cc.	Polarization after 5 days.	Polarization after 8 days.	Polarization after 14 days.
30	..	7.1	3.6
40	7.6	7.1	4.4
60	..	8.0	4.6

We were unable to clarify milk in such a manner that the lactose would not be consumed by the organisms present in pressed yeast even in the presence of potassium fluoride. It was next attempted to add to the clarified milk something which would feed the yeast and give it such a start that it might be able to assist in holding the lactic acid ferment in check. Cane sugar seemed to be best adapted for this purpose. A sample of milk was taken and divided into portions of 100 cc. each. To each portion was added ten grams of cane sugar dissolved in fifty cc. of water, one-half cake of yeast and an amount of fluoride varying from six to forty mgms. One portion of the milk was clarified and polarized at the beginning of the operation without the addition of sucrose. The portions fermented were polarized on the eighth, eleventh, and thirteenth days. As will be seen in table 29, the original polarization of the lactose was not obtained in any case.

TABLE 29.

Sucrose, grams.	Mgms. KF. per 100 cc.	Total liquid in cc.	Original polarization lactose.	Polarization after 8 days.	Polarization after 11 days.	Polarization after 13 days.
10	6	150	14.4	10.8	10.8	8.4
10	11	150	14.4	11.8	12.0	10.6
10	17	155	14.4	14.0	12.8	11.0
10	23	155	14.4	....	13.8	12.4
10	40	165	14.4	....	....	....

Another portion of milk was sterilized by boiling, allowing to stand for four hours, and again boiling. It was then treated just as in the previous experiment with the same result. See table 30.

TABLE 30.

Sucrose, grams.	Mgms. KF, per 100 cc.	Total liquid in cc.	Original polarization lactose.	Polarization after 5 days.	Polarization after 9 days.	Polarization after 14 days.	Polarization after 20 days.
2	40	135	14.4	12.4	10.6	6.4	....
2	40	135	14.4	12.4	11.0	4.8	....
10	0	150	14.4	....	12.2	8.6	6.6
10	40	160	14.4	....	12.8	11.6	6.6

Two liters of milk were then clarified by heating to 80° for one-half hour with twenty cc. of glacial acetic acid, filtered, almost neutralized with potassium hydroxide, again filtered, and cooled to the temperature of the room. This filtrate was divided into several portions, and each portion diluted more or less with water. Each of these was again subdivided into portions of 100 cc. each, and to each of these was added a solution of ten grams of cane sugar in water, one-half cake of yeast, and an amount of fluoride varying from 0 to 100 mgms. per 100 cc. of the solution. Similar portions, of course, were polarized without the addition of sucrose. The result was still unsatisfactory, for before the fermentation of the sucrose was complete the milk sugar had begun to decompose. The results are given in table 31.

TABLE 31.

Sucrose, grams.	Mgms. KF, per 100 cc.	Total liquid in cc.	Original polarization.	Polarization after 8 days.	Polarization after 18 days.	Polarization after 20 days.
10	0	150	10.8	9.4	....	....
10	7	150	10.8	10.4	6.0	4.4
10	13	150	10.8	10.8	....	5.3
10	20	155	10.8	10.4	....	....
10	27	155	10.8	10.1	....	....
10	33	160	14.2	13.0	9.0	....
10	40	160	14.2	12.4	....	....
10	47	160	14.8	12.3	7.2	1.8
10	53	165	14.8	12.4	5.5	....
10	60	170	14.8	12.6	6.0	....
10	70	170	9.4	8.6	5.4	1.6
10	80	175	9.4	7.6	5.2	....
10	100	180	9.4	7.9	....	....

A liter of milk was then clarified with oxalic acid, filtered, neutralized, filtered, measured out in portions of 100 cc. each, ten grams of cane sugar added to each portion together with one-half cake of yeast, and a varying amount of fluoride. The

solutions were polarized after eight and fourteen days. On the eighth day the fermentation of the sucrose seemed to be complete, and no decomposition of lactose was evident. On the fourteenth day, however, the filtrates were very dark and the readings could only be taken with difficulty. There was also a very marked decrease in the amount of lactose present. The results are given in table 32.

TABLE 32.

Sucrose, grams.	Mgms. KF, per 100 cc.	Total liquid in cc.	Original polarization.	Polarization after 8 days.	Polarization after 14 days.
10	13	150	9.9	10.0	5.1
10	20	155	9.9	9.9	6.8
10	27	155	9.9	9.9	7.4
10	33	155	9.9	9.8	8.8

Two liters of milk were then heated to 80° on the steam bath, and a solution of eight grams of glacial phosphoric acid added and the whole kept at this temperature for one-half hour. At the end of this time it was filtered, neutralized, and filtered again. Portions of 100 cc. each were then measured into 200 cc. flasks, and to each was added a solution of cane sugar containing either five or ten grams of sucrose, as shown in the table, and one-half cake of compressed yeast. A portion of the filtrate was also polarized, and gave a reading of 14.4° on the sugar scale. The flasks which were set aside for fermentation were polarized, as is shown in table 33, after eight, ten, fourteen, and eighteen days.

TABLE 33.

Sucrose, grams.	Mgms. KF, per 100 cc.	Total liquid in cc.	Original polarization.	Polarization after 8 days.	Polarization after 10 days.	Polarization after 14 days.	Polarization after 18 days.
5	30	125	14.4	...	14.4	...	14.0
5	30	125	14.4	...	14.2	14.2	...
5	40	125	14.4	...	14.6	...	14.4
10	40	150	14.4	14.4	...	14.3	...
10	40	160	14.4	14.4	...	14.3	...
10	53	160	14.4	14.4	...	14.3	...

As before stated, any solution whose polariscopic reading was desired was first diluted to 100 cc. or 200 cc. In those cases in which 200 cc. was polarized the reading was multiplied by two, so that in the reading given it is always supposed that the amount of sugar present is dissolved in 100 cc. of water. It is

found that the volume of yeast was not great enough to cause any error. The fermentation of sucrose dissolved in milk whose casein had been precipitated by means of phosphoric acid was repeated several times with the same result in every instance. Whenever five to ten grams of sucrose were present at the beginning of the operation, the sucrose was completely fermented at the end of eight to ten days, while the lactose had not been affected at the end of two weeks.

The details of the application of these methods to the determination of the sugars in condensed milk are as follows:

The entire contents of the can are transferred to a porcelain dish and thoroughly mixed. A number of portions of about twenty-five grams are carefully weighed into 100 cc. flasks. Water is then added to two of the portions, and the solutions boiled to make sure that all the lactose is in solution and possesses the normal rotation. The flasks are then cooled, clarified by means of mercuric iodide dissolved in acetic acid, and alumina cream, made up to mark, filtered and polarized. Other portions of the milk are placed in a water bath and heated to the temperature of 55°. One-half cake of compressed yeast is then added to each flask and the temperature maintained at 55° for five hours. Mercury solution and alumina cream are then added, and the solution is cooled to room temperature, made up to the mark, mixed by shaking, filtered and the filtrate polarized. The amount of cane sugar present is then determined by Clerget's formula. In each case the volume of precipitated milk solids may be estimated by the double dilution method. The reducing sugar is then estimated by one of the copper reducing methods. If the amount of reducing sugar estimated as milk sugar, plus the amount of cane sugar obtained by inversion, would give a polarization equal to that obtained by the direct reading of both sugars before inversion, it is evident that no invert sugar is present, and is not necessary to test farther. If, however, the amount of reducing sugar seems to be too great, invert sugar is probably present, and the milk sugar must be determined, by another method. For this purpose 250 grams of condensed milk are dissolved in water and the solution boiled to secure the normal rotation of the milk sugar. The solution

is then allowed to cool to about 80°, a solution of $3\frac{1}{2}$ to 4 grams of glacial phosphoric acid in water is then added, and the mixture kept at this temperature for a few minutes. The mixture is then cooled to room temperature, filled up to the mark with water, thoroughly mixed by shaking and filtered.

It may be assumed that the volume of the precipitate is equal to that obtained by precipitation with mercury solution. Enough potassium hydroxide is then added to almost but not quite neutralize the free acid, and sufficient water is added to make up for the volume of the solids precipitated by the phosphoric acid. The mixture is then filtered and the filtrate is measured in portions of 100 cc. each, into 200 cc. flasks. A solution containing about twenty mgms. of potassium fluoride, and a half cake of Fleischman's compressed yeast, are then added to each flask and the mixture is then allowed to stand for ten days in a room whose temperature is 25° to 30°. The flasks are filled up to the mark, and the milk sugar is determined either by copper reduction or by means of the polariscope.

Invert Sugar.—The weight of cuprous oxide reduced by milk sugar and invert sugar, less the equivalent of the milk sugar found after fermentation, is due to invert sugar.

CHEMICAL CONSIDERATIONS ON THE POTTERY INDUSTRIES OF THE UNITED STATES.

By Karl Langenbeck.

Received November 27, 1893.

PART II.

Rockingham and Yellow Ware.—A higher grade of ware, than that last treated is "Rockingham and Yellow Ware;" though belonging to the same ceramic category, namely, "Faience," in that it also consists of a porous body covered with a transparent lead glaze.

It differs from "red ware" in color, and in having a body requiring a higher temperature for its proper burning, so that the glaze is not applied to the freshly formed pieces in their "clay state," but to the once baked or "biscuit" pieces, and is then finished in a second or "glost" fire softer than the first.

The clays used for "yellow ware" belong to the class commercially known as second-class fire clays; the same from which common fire brick and such terra-cotta articles as stove and flue linings, chimney tops, garden vases, etc., are made. They are generally the common "buff" or "blue" clays of the coal measures and are widely distributed in all our carboniferous exposures.

A typical clay of this character has the following composition:

TOTAL ANALYSIS.

	Per cent.	Insol. in H_2SO_4 Per cent.
Silica	60.50	22.33
Alumina	25.53	0.53
Ferric Oxide	1.66	0.26
Titanic Oxide	0.54	....
Manganous Oxide	0.33	....
Lime	0.38	0.26
Magnesia	1.19	0.07
Alkalies	1.76	0.37
Combined Water	7.98	....
	99.87	23.82

RATIONAL ANALYSIS.

	Per cent.
Clay Substance	76.05
Quartz	19.54
Feldspathic detritus	4.28
	99.87

CHEMICAL COMPOSITION OF THE "CLAY SUBSTANCE."

	Per cent.
Silica	50.19
Alumina	32.87
Ferric Oxide	1.84
Titanic Oxide	0.71
Manganous Oxide	0.43
Lime	0.16
Magnesia	1.47
Alkalies	1.82
Combined Water	10.50

The yellow ware potter is better equipped in machinery than

the red ware potter, and does not, like the latter, prepare his clay by merely soaking it with water and then tread and knead it to a plastic mass of the proper consistence; but subjects the clay to a regular washing process. Hence the presence of a certain amount of coarse sand and nodules and particles of iron pyrites, very commonly found in all such clays, do not spoil them for his work, as they are removed by the necessary and customary process of manufacture.

This consists in "slipping" the clay in a vat with mechanical stirrers, known as a "blunger," sifting the "slip" through a sixty-mesh wire sieve or a No. 8 silk lawn stretched over a vibrating frame, from which the coarser sandy impurities are thrown, and condensing the "slip" to plastic clay by evaporation or by a filter press.

From this process, it will be realized that the clay must be in such a condition of physical aggregation as to be easily disintegrated or "slipped" by mere stirring in water. A hard clay readily reduced by the elements in "weathering" to such a condition, will also answer the purpose; but the double shoveling involved in transferring clay from the bank to a weathering flat, and from thence, after six months or a year's exposure to the elements, to the factory, is a trouble the potter avoids if he can.

There is a class of clays, which from their composition are admirably adapted for yellow ware, but have remained completely barred from this use because of their physical character. These are the "flint clays" of which the following analysis is typical:—

TOTAL ANALYSIS.

	Per cent.	Insol. in H_2SO_4. Per cent.
Silica	55.04	16.96
Alumina	29.85	0.11
Ferric Oxide	1.76	....
Lime	0.79	0.12
Magnesia	0.57	0.17
Alkalies	1.83	0.58
Combined Water	10.95	....
	100.79	17.94

RATIONAL ANALYSIS.

	Per cent.
"Clay Substance"	82.85
Quartz	16.58
Feldspathic detritus	1.35
	100.78

PERCENTAGE COMPOSITION OF THE "CLAY SUBSTANCE."

	Per cent.
Silica	45.97
Alumina	35.90
Ferric Oxide	2.12
Lime	0.81
Magnesia	0.48
Alkalies	1.51
Combined Water	13.22
	100.00

This clay occurs in rocky masses having a conchoidal fracture, the splinters of which are so hard and sharp, that a flying piece, struck off with the pick, will cut the hand or face. It eagerly absorbs water and with a crackling noise, though without noticeable evolution of heat, falls to pieces, yielding a mass of shell-like splinters, though a year's exposure to the weather, while reducing it to a fine sand, fails to produce a workable clay. Simple grinding in water and thickening the resulting "slip," by any convenient method, produces a highly plastic mass, that is readily formed into ware, burns to a bright yellow color, and bears the customary yellow ware glazes with less danger of "crazing" than the before-mentioned and commonly used clay. This latter fact is to be accounted for by the extreme fineness of the contained quartz, more than compensating for the reduced quantity of the same.

The expense of wet grinding a "flint clay" would not be excessive, but the introduction of machinery for the purpose, in so conservative a craft as the one under consideration, would be met with almost stolid resistance.

The first or "biscuit" fire of yellow ware, for hardening the clay pieces, must reach "good biscuit heat," that is a temperature sufficient to bake the clay so hard that it can no longer be cut with a knife, but that the steel leaves a lead-pencil-like

mark on the surface. At the same time, the shard must still be porous and adhere when touched to the tongue.

The temperature of the "biscuit" fire will necessarily, of course, vary with the chemical and physical character of the clay used. The potter determines it empirically by testing-trials of the clay drawn from the kiln, with his knife and tongue.

It is very important that the chemist determine at what heat the clay he is examining attain a "good biscuit" condition, in order that the potter may know whether he can introduce ware made of it into his biscuit kiln along with his old ware. As a kiln of ware represents considerable capital exposed to a risky operation, potters naturally are absolutely opposed to the adoption of new clays requiring different conditions of fire from those already adopted by them; these must dovetail easily at least into their kiln conditions, by requiring the same heat.

For the purpose of such heat tests the most accurate and convenient appliance is the Seger pyrometric cone. The cones are easily used, not influenced by momentary flashes of unequal heats, but melt down at the corresponding average temperature of a certain period of time (which is what determines the character and hardness of the baked product); they accord perfectly with others of the same number, are cheap and if not easily obtained can even be made by the operator himself.

Yellow ware clays reach the required hardness at temperatures varying from the melting points of cones V, to VII, or even VIII.[1]

		Per cent.
[1] V. $\left.\begin{matrix}0.3\ K_2O\\0.7\ CaO\end{matrix}\right\}$ $0.5\ Al_2O_3$ $5.\ SiO_2$	Orthoclase	83.55
	Marble dust	35.00
	Quartz	84.00
	Kaolinite	25.90
VI. $\left.\begin{matrix}0.3\ K_2O\\0.7\ CaO\end{matrix}\right\}$ $0.6\ Al_2O_3$ $6.\ SiO_2$	Orthoclase	83.55
	Marble dust	35.00
	Quartz	108.00
	Kaolinite	38.85
VII. $\left.\begin{matrix}0.3\ K_2O\\0.7\ CaO\end{matrix}\right\}$ $0.7\ Al_2O_3$ $7.\ SiO_2$	Orthoclase	83.55
	Marble dust	35.00
	Quartz	132.00
	Kaolinite	51.80
VIII. $\left.\begin{matrix}0.3\ K_2O\\0.7\ CaO\end{matrix}\right\}$ $0.8\ Al_2O_3$ $8.\ SiO_2$	Orthoclase	83.55
	Marble dust	35.00
	Quartz	156.00
	Kaolinite	64.75

[1] *Thonindustrie Zeitung*, 1886, p. 169.

For "yellow ware" as for "red ware," it is customary to employ a "raw" glaze, that is, one containing no soluble constituents which have first to be rendered insoluble by fritting to a glass.

The customary type of glaze has a formula resembling one of the following: 1.0 PbO 0.2 Al_2O_3 2.0 SiO_2, which may be made by grinding together:

129.7 White Lead
25.9 China Clay
48.0 Flint

0.8 PbO
0.2 K_2O
} 0.2 Al_2O_3 2. SiO_2 from

1.0

103.8 White Lead
55.7 Feldspar
24.0 Quartz

0.8 PbO
0.1 CaO
0.1 K_2O
} 0.2 Al_2O_3 2. SiO_2

1.0

obtained by grinding together:

103.8 White Lead
27.9 Feldspar
13.0 China Clay
5.0 Carbonate of Lime
36.0 Quartz

The acidity of the glaze generally varies from 1.8 to 2.2 SiO_2, depending upon the amount of quartz contained in the clay upon which it is expected to stand. The alumina will similarly vary from 0.16 to 0.2 and over according to the stiffness of the glaze required and the conditions of firing, causing a liability to devitrification, or most likely as chance has thrown a fairly satisfactory formula into the potter's hands.

The temperature at which the glazes run bright lies about at a point at which an alloy composed of

50 per cent. Silver
50 " " Gold

will melt, though depending upon the composition of the glaze, and the length of fire, the melting point of the alloy,

75 per cent. Gold
25 " " Silver

may have to be reached.

"Rockingham ware" differs from "yellow ware" only in that it is covered with a brown manganiferous glaze, applied either by spattering the piece, previously dipped in the clear glaze, with the same, thus producing a mottled effect by the melting of the glazes into each other, or by directly dipping the biscuit piece into the "Rockingham" glaze alone, whereby the fired piece obtains a uniform red-brown finish.

Common forms of Rockingham glazes are the following:

$$\left.\begin{matrix} 0.85\ PbO \\ 0.15\ MnO \end{matrix}\right\} 0.18\ Al_2O_3\ 1.8\ SiO_2$$
$$1.00$$

made by grinding together:

110.0 White Lead
6.5 Black Oxide of Manganese
23.3 China Clay
43.2 Flint

also,

$$\left.\begin{matrix} 0.9\ PbO \\ 0.1\ MnO \end{matrix}\right\} \left.\begin{matrix} 0.15\ Al_2O_3 \\ 0.05\ Fe_2O_3 \end{matrix}\right\} 2.\ SiO_2$$
$$1.0$$

similarly prepared with a ferruginous clay, the formula of which any chemist will be able to figure out from the equivalent proportions given.

As in the case of "red ware" the prevailing fire of both the biscuit and glost kilns should be oxidizing.

The ware manufactured in this industry embraces such kitchen and other domestic utensils as bowls, bakers, nappies, chambers, tea and coffee pots, pitchers, etc.

In examining a clay for its possible use in this manufacture it is important to determine whether it can readily be disintegrated by stirring or boiling in water, or whether repeated soaking and drying or freezing and thawing it bring it to such a state.

A "slip" of the clay should be run through a sixty mesh wire sieve and the amount and character of the remaining sand given.

If the latter is at all appreciable the sample for analysis should not be taken from the crude clay, as this would, of course, give an entirely false idea of the constituents ultimately entering the potter's "body," but the sample should be taken from the thickened and dried "slip" which has passed the sixty mesh sieve. Such a "slip" is readily thickened by pouring it on a thick, clean, and dry slab of plaster paris, which readily absorbs the water and from which the plastic clay is readily peeled without danger of contamination with plaster.

A part of the now plastic clay is dried for preparation of the sample for a "rational" analysis, in the customary manner. The bulk of the washed product is formed into cakes and rings for the burning. The latter are made for the purpose of watching the progress made by the clay in the fire, and determining when the baking is finished. These rings should be of such size, as to be readily withdrawn from the muffle with a stout iron wire, through the spy-hole in the door of the same.

When the clay pieces are bone-dry, they are placed in the muffle on a thick bed of clean quartz sand or a thick fire-clay tile strewn with the same.

The rings should be so placed as to be easily reached through the spy-hole in the door brick and parallel with them the Seger cones V, VI, VII, and VIII, should be set upright on a piece of tiling or flat cake of baked or raw clay, to which they had best be stuck with a little "slip." The door brick is then luted in place with a wad of soft clay, and the firing begun gradually, raising the heat at an increasing rate.

Should there be difficulty in seeing the pyroscopes when the muffle has reached bright redness, on removing the plug from the spy-hole, the careful introduction of a thick iron wire in their

neighborhood, will momentarily so far reduce their temperature as to make them clearly discernible against the bright walls of the muffle.

When cone V crooks over, the first clay ring should be drawn from the muffle with a wire and when cool, tested with the knife for hardness, if insufficient the firing is continued until cone V has melted down completely, when the second ring is drawn. The next trial is drawn at the crooking of cone VI, and so on until a heat is reached at which the clay trial ring is sufficiently hard to resist cutting with a knife, without having lost all porosity, adhering when touched to the tongue.

When this heat is reached the firing is discontinued and the furnace allowed to cool.

The fired pieces of clay removed from the cold muffle are covered on one side with one of the described glazes, of which I would recommend the first, which is easily made and fired as a convenient empirical standard, against which to test the coefficient of expansion of the baked clay.

The pieces are then baked a second time to melt the glaze upon them, the heat reached being that of the melting point of the alloy,

50 per cent. Silver,
50 " " Gold.

It is also very convenient to use the small baked trial rings partly covered with the glaze as trials for this fire, the firing being discontinued when the glaze on one of these rings drawn from the muffle has run bright.

The color of the clay under the clear glaze should be described. Tints approaching bright straw and lemon yellows are the ones sought after. Brownish and reddish tints are not acceptable to the trade buying the ware.

The behavior of the glaze on the clay must also be given. From these data the potter will be able to determine whether the acidity of his glaze must be increased or diminished, or which clays to mix, if he chooses to keep the glaze as it is and compensate the incorrect coefficient of expansion of the clays by mixing them.

NOVEMBER 25, 1893.

THE SYNTHESIS OF POLYHYDRIC ALCOHOLS BY MEANS OF FORMALDEHYDE.[1]

By H. Hosäus, P. Rave, P. Wigand, and B. Tollens.

I. INTRODUCTION.

By B. Tollens.

THE aldehyde of formic acid or of methyl alcohol, the formaldehyde CH_2O, which can be easily obtained in large quantities, and which at present is assumed to furnish the sugars in the vegetable kingdom by polymerization, is a very favorable material for the synthetic formation of polyhydric alcohols.

Formaldehyde possesses the property when added to other aldehydes or ketones in the presence of calcium or barium hydroxide of combining directly with these, forming compounds in which one or more primary alcohol groups (methyl according to the Geneva nomenclature) are present.

According to the experiments carried out by the above gentlemen and myself the carbon atoms combined with the aldehyde group COH or the ketone group CO of the bodies acting upon formaldehyde react by combining with as many CH_2O groups as there are hydrogen atoms united to them.

Probably formaldehyde and water form methylen glycol $CH_2(OH)_2$ and this condenses with the aldehydes or ketones with the separation of OH and H or water.

Thus CH_2 and $2CH_2O$ form $C(CH_2OH)$ and CH_2 with $3CH_2O$ forms $C(CH_2OH)_3$.

At the same time the aldehyde group COH becomes CH_2OH and the ketone group CO becomes CHOH. There are therefore two more atoms of hydrogen taken up, another portion of formaldehyde forming formic acid with the decomposition of a molecule of water.

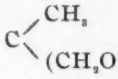
As a result of these changes the following reaction takes place in the case of acetaldehyde:

$$\begin{matrix} CH_3 \\ | \\ COH \end{matrix} + 4CH_2O + H_2O = \begin{matrix} C-(CH_2OH)_3 \\ | \\ CH_2OH \end{matrix} + CH_2O_2$$

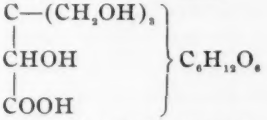
[1] Read before the World's Congress of Chemists, August 23, 1893.

a body being formed which contains four CH_2OH groups combined with one carbon atom or $C(CH_2OH)_4$.

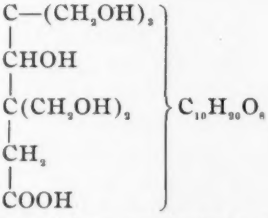
Propionaldehyde, CH_3CH_2COH in which the carbon atom which is linked to the aldehyde group is combined with only two hydrogen atoms, forms CH_3, $C(CH_2OH)_2$, CH_2OH or



Pyrotartaric acid $CH_3.CO.COOH$ forms

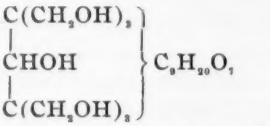


Levulinic acid, $CH_3.CO.CH_2.CH_2.COOH$, combines, as it contains a carbon atom with three hydrogen atoms and another with two hydrogen atoms linked to CO groups, with $5CH_2O$ groups forming



The bodies thus formed from pyrotartaric and levulinic acids with formaldehyde are on account of the presence of the carboxyl group still acid and are further decomposed with the separation of water forming lactones or anhydrides. Thus pyrotartaric acid gives $C_6H_{10}O_5$ and levulinic acid $C_{10}H_{16}O_6$. (See below).

Acetone, $CH_3.CO.CH_3$, probably takes up $6CH_2O$ groups and forms



or better according the analyses so far made $C_9H_{18}O_6$ a hepta-

tomic alcohol with nine carbon atoms which is partially an anhydride.

The reaction of formaldehyde upon aldehydes and ketones above described is probably general in the alcohol and fatty acid series and we are at present engaged in a further investigation which will no doubt furnish a greater number of polyhydric alcohols and alcohol acids.

II. DESCRIPTION OF THE PRODUCTS ABOVE MENTIONED.

(*a*) *Pentaerythrol*, $C(CH_2OH)_4$. (*Tetramethylolmethane*).

By B. Tollens, P. Wigand, and P. Rave.

This very interesting compound, which was first prepared some years ago[1] but has been investigated only lately crystallizes very beautifully and is formed by mixing

600 grams formaldehyde (32.4 per cent.),
60 grams acetaldehyde,
160 grams calcium hydroxide,
and 9 liters water.

The mixture is allowed to stand several weeks with frequent shaking. The liquid is then decanted, the calcium removed by precipitation with oxalic acid and upon evaporation and separation of the syrup from the crystals by filtration about 115 grams of crude pentaerythrol are obtained.

This is purified by repeated crystallization and extraction with alcohol and then melts at 250–255°. It shows all the properties of a tetrahydric alcohol with five carbon atoms, so that we chose the above name for it as an empirical homologue of erythrol.

As tetrahydric alcohol it forms a tetracetate and tetrabenzoate, with hydrobromic acid and a tri-and tetrabromhydrin, with hydriodic acid a di-, tri-, and tetraiodhydrin which all crystallize well and show definite melting points.

With nitric acid and with chromic acid it is decomposed and furnishes among other products glycolic acid but no acetic acid. With iodine and sodium hydroxide it does not form iodoform.

From these reactions and the fact that it does not form a secondary iodide with hydriodic acid (as for instance mannitol forms secondary hexyl iodide) we reason that the carbon atoms

[1] *Annalen der Chemie*, 265, 315; 276, 58.

in pentaerythrol are not regularly linked but that it possesses the structure of a methane in which the four hydrogen atoms are replaced by four CH_2OH groups.

(*b*) *Pentaglycerol* $C\begin{matrix}CH_3\\(CH_2OH)_3\end{matrix}$ (*Methyltrimethylolmethane*).

By H. Hosäus.

When

20 grams propionaldehyde,
80 grams formaldehyde (40 per cent.),
1100 grams water,
and 50 grams calcium hydroxide

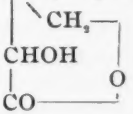
are heated on the water bath, pentaglycerol is formed.

$$\begin{matrix}CH_3\\|\\C(CH_2OH)_2\\|\\CH_2OH\end{matrix} \quad \text{or} \quad C\begin{matrix}CH_3\\(CH_2OH)_3\end{matrix}$$

The liquid is decanted, the dissolved lime precipitated with oxalic acid and the filtrate evaporated to a syrup which slowly crystallizes. The crystals show after purification the melting point 199° and prove to be a trihydric alcohol with five carbon atoms. Pentaglycerol upon distillation in vacuo yields at 165° a triacetate and a tribenzoate.

When oxidized with chromic acid it yields acetic acid and this proves that it contains a methyl group.

(*c*) *Lacton from pyrotartaric acid and formaldehyde.* (*Trimethylol lactic acid lacton*).



By H. Hosäus.

By warming

27 grams pyrotartaric acid,
90 grams formaldehyde (40 per cent.),
70 grams calcium hydroxide,
and 1450 grams water

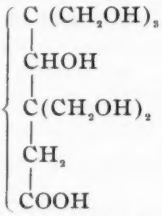
there is formed the calcium salt of a lactic acid in which three hydrogen atoms are replaced by three CH_2OH groups and by precipitation of the calcium with oxalic acid and evaporation of

the filtrate this acid is changed into its lacton. After purification the crystals melt at 184°.

This lacton which is neutral regenerates the acid when boiled and combines with 1 molecule of NaOH or $\frac{1}{2}$ molecule of CaO. It does not react with iodine and sodium hydroxide nor with phenylhydrazin and therefore does not contain any pyrotartaric acid.

(*d*) *Lacton from levulinic acid and formaldehyde.*

Anhydride of



* By P. Rave and B. Tollens.

We obtained the barium salt of this acid by allowing

50 grams levulinic acid,
250 grams formaldehyde (40 per cent.),
200 grams crystalline barium hydroxide,
and 5 liters water

to stand fourteen days with frequent shaking, then heating, carefully precipitating the barium with sulphuric acid and evaporating the filtrate. From the syrup we obtained the lacton $C_{10}H_{16}O_8$, crystallizing in small plates melting at 174–176°, the formula being controlled by Raoults method.

This compound reacts neither with iodine and sodium hydroxide nor with phenylhydrazin therefore containing no levulinic acid. It is almost neutral but combines with one molecule of NaOH on boiling. It is derived from hydrogenized levulinic acid or hydroxy-valerianic acid, being anhydro-pentamethylol-hydroxy-valerianic-acid-lacton and formed from the above acid by the loss of $2H_2O$.

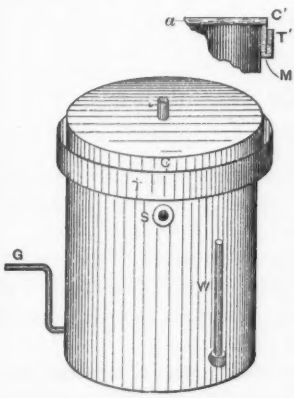
When of the six hydroxyls contained in the above formula one forms a lacton by combining with the carboxyl with separation of H_2O and two others unite in a manner similar to an ether, only three hydroxyls remain, and it is a fact that upon heating this lacton with acetic anhydride three acetyl groups are introduced, a well crystallizing triacetate of the lacton being formed.

A DRYING OVEN FOR DRYING IN HYDROGEN AT THE TEMPERATURE OF BOILING WATER.

By F. W. Morse.

Received September 28, 1893.

THE following described oven has been in use in the laboratory of the New Hampshire Experiment Station for over a year, and proves to be efficient for drying substances in an atmosphere of hydrogen at the temperature of boiling water. It consists of a cylindrical copper box with double bottom and wall. The inside of the oven is seven inches in diameter and nine inches in depth. The space between the walls is one inch. It is fitted with a water-gauge, W, and a steam outlet, S. This outlet is a screw-nipple, and may be coupled to a condenser if desired. The hydrogen enters the oven by means of a brass tube, G, which is coiled in the space between the bottom and wall and enters the inner oven near the top. The gas is thus thoroughly heated before entering the drying compartment. The gas passes out of the oven near the bottom by an outlet not shown in the cut. The oven is made gas-tight by a mercury seal. A copper trough, T T' is fitted around the top of the oven a little below the rim. The trough is one and one-fourth inches in depth and one-half inch in width, and is made with brass joints and coated on the inside with lacquer. The cover, C C', fits loosely in the trough, and the mercury, M, makes a perfectly tight joint. Drying is hastened by placing an acid dish containing concentrated sulphuric acid on the bottom of the oven. A rack rests upon the acid dish to receive the watch glasses or drying flasks.



In addition to heating the gas, the oven is made more efficient by blackening the inner walls to increase radiation, and

lining the under side of the cover with a thick sheet of asbestos. The outside of the cover is plated with nickel which diminishes radiation outward. Repeated trials with a standard thermometer inserted in the oven at *t*, and between the walls at S, have shown a difference of only two-tenths of a degree between the boiling water and the drying compartment. Trials with several ordinary chemical thermometers have shown a variation from 99° C. to 101° C. in the oven.

The oven was made for the Experiment Station by Richards & Co., of New York.

NEW HAMPSHIRE EXPERIMENT STATION,
DURHAM, N. H.

AN IMPROVED GOOCH CRUCIBLE.

BY W. A. PUCKNER.

Received November 14, 1893.

SOMETIME ago I adopted a slight improvement in the use of the Gooch crucibles with which I am so well pleased that I wish to call attention to it.

It is an occasional, if not frequent, occurrence, that while pouring a precipitate on the asbestos, this becomes loose, and floats in the liquid, thus allowing the precipitate to pass through the perforations.

Another objection, which is sometimes urged against this method of filtration, is that the asbestos becomes mixed with the precipitate making an examination regarding purity difficult.

My modification consists in placing a perforated platinum plate, such as are used in the Gooch crucibles made of porcelain, and which may be obtained separately, on the layer of asbestos. This layer need be but very thin as there is now not the least danger of disarranging it while pouring on the liquid. After drying or ignition the precipitate can be obtained perfectly clean and with the loss of but a few milligrams.

HEIDELBERG UNIVERSITY.

PATENTS OF INTEREST TO CHEMISTS.

EDITED BY ALBERT H. WELLES.

Ore Separators, etc.—508,679, November 14, Carter, C., stamp mill and amalgamator. 508,008, November 7, Gelien, G. A., ore grinder. 508,542, November 14, Greer, R. C., ore roasting furnace. 508,381, November 7, Giroux, J. L., smelting furnace. 509,450, November 28, Rushforth, A. P., cupola or blast furnace. 509,439, November 28, Mace, G. A., lime-kiln. 509,550, November 28, Nickerson, T. K., lime-kiln. 508,282, November 7, Clancy, W., apparatus for screening and concentrating ores. 509,818, November 28, Lockhart, W. S., washing and separating minerals, jigging apparatus.

Gold and Silver.—509,289, November 21, Bohm, W. D., apparatus for separation of gold from ores. 509,368, November 28, Engelhardt, E. C., treatment of ores; bromine, hydrochloric acid, and sodium carbonate are reagents used.

Lead.—508,115, November 7, Noad, J., white lead and colored pigments; float lead is treated with a solution of glycerol and acetic acid and carbon dioxide is admitted. 509,057, November 21, Waller, E., white lead; native lead carbonate heated with ammonium acetate solution is distilled, ammonium carbonate is condensed, and solutions of this and lead acetate are mixed. 509,058, November 21, Waller, E., and Sniffin, C. A.; argentiferous lead carbonate ores are concentrated by dissolving lead in acetic acid and boiling neutral acetate solution to convert into basic lead acetate, while silver is concentrated in the insoluble residue. 509,059, November 21, Waller, E., and Hinman, B. C.; white lead is precipitated from a basic lead acetate solution by dissolving carbon dioxide in water under pressure and forcing basic acetate into tanks.

Copper.—509,619, November 28, Helbig, P., hardening copper; melted copper is treated with glass and subcarbonate of iron and antimony is added. 509,633-634, November 28, Tuttle, D. K., and Whitehead, C.; slimes from refined copper are treated with dilute acid then with sulphuric acid or silver sulphate.

Aluminum.—508,796, November 14, Ackerman, W., aluminum fluoride; aluminum silicate is treated with hydrofluoric acid.

Antimony.—509,478, November 28, Mayer, T.; antimony oxide is dissolving in thirty-three per cent. hydrochloric acid and sixty-seven per cent. hydrofluoric acid and an alkali sulphate is added forming $2SbF_3$, M_2SO_4, readily soluble in water.

Acids and Alkalies.—509,664, November 28, Howard, H., automatic regulator for concentrating sulphuric acid. 508,804, November 14, Blackmore, H. S., electrolysis of salts of alkalies. 508,915, November 21, Cardozo, D. P. F., manufacture of salt; jets of air are forced into brine to concentrate it, the liquid is then transferred to another tank to deposit im-

purities, and then to a third tank and evaporated by air. **508,241**, November 7, Richardson, J. C., electrolytic apparatus for the fixed caustic alkalies from sodium and potassium chlorides. **509,163**, November 21, Helmer, N., ozone; prepared by adding hydrogen peroxide to a potassium carbonate solution.

Fertilizers.—**508,220**, November 7, Greenstreet, C. J.; nitrogenous fertilizer; tank water or stock is combined with a salt of manganese or iron.

Sanitary Chemistry.—**509,560**, November 28, Sprague, A. V. M., sterilizing apparatus.

Brewing and Distilling.—**508,882**, November 14, Hornbostel, C.; fermented and distilled liquors are treated with kieselguhr. **508,913**, November 21, Burkhardt, A. G., apparatus for distilling and rectifying.

Bleaching and Dyeing.—**508,261**, November 7, Weldon, L., apparatus for dyeing. **507,995**, November 7, Clegg, C. T., apparatus for bleaching and dyeing. **509,431**, November 28, Graemiger, A. and J., apparatus for dyeing. **508,592**, November 14, Austen, P. T., friable coloring matter obtained from dye-wood extracts by incorporating ammonium carbonate. **509,703**, November 28, Taylor, A., extract from red-wood tree by treating with water and caustic alkali, neutralizing and precipitating solid matters. **509,635**, November 28, Ulrich, M., and Bammann, J., brown tetrazo dye. **509,623**, November 28, Lauch, R., brown dye, $C_{13}H_9N_3O_6$; made by treating with nitrous acid the compound resulting from action of diazotized amido hydroxy carbonic acid on resorcinol, or orcinol.

Tanning.—**508,281**, November 7, Chailly, F., tanning apparatus.

Organic Compounds.—**509,087**, November 21, Majert, W., piperazine. **509,055**, November 21, Thoms, H., salicylate of orthotolyl dimethylpyrazolon, $C_{12}H_{14}N_{20}.C_7H_6O_3$. **509,520**, November 28, Fritsch, Paul, salicylic ester of acetol; made by heating a salicylate of an alkali and monohalogen acetone and separating the alkaline chloride from acetol formed. **508,124**, November 7, Turgard, H. D., denitrating nitro-cellulose by immersing in ammonium hydrosulphate and a metallic sulphide.

Sugar.—**509,749**, November 28, Morrell, J. A., process of and apparatus for evaporating cane juice. **508,747**, November 14, Pellegrini, J. E., synthetic sugar; four parts of carbon dioxide, four parts of ethylene, and three parts of water are united by osmose.

Oils and Varnishes.—**508,479**, November 14, Kayser, A., hydrocarbon oils are deodorized by treating dried vapors with anhydrous nitric acid.

Plaster and Cement.—**508,033**, November 7, Mohle, A., artificial stone; sulphuretted slag, brick dust, waste sandstone, sand, cement, and sodium carbonate are used. **508,731**, November 14, Majewski, H. A., artificial marble made from calcium sulphate, potassium sulphate, and a solution of alum. **509,428**, November 28, Edison, T. A., composition brick; powdered rosin and petroleum are added to ore and heated to expel petroleum, while part of the rosin combines with the metallic oxide to form a resinate.

Miscellaneous.—**509,396**, November 28, Pennington, John C., retting bath for flax, composed of water, caustic potash, ammonia, sodium phosphate, magnesium sulphate, and manganese chloride. **508,015**, November 7, Hoffman, H., removing bitterness from kola powder by treatment with hydrogen peroxide. **509,617**, November 28, pharmaceutical compound; made from three parts of iodine and one part of meta cresotinic acid. **508,608**, November 14, Etheridge, R. L., rosin, made by distilling crude turpentine and bluing. **509,209**, November 21, Draper, J., scale is removed from boilers by a composition of water, mercury, sodium, or potassium, and sodium carbonate under heat and pressure. **509,280**, November 21, Ash, T. M., to coat non-metallic articles; first cover, with a carrying medium and finely divided metal then treat with silver nitrate. **508,560**, November 14, Briersdorf, P. C., treatment of gutta percha, treating with solvent to obtain uniform qualities. **509,460**, November 28, Weinrich, M., revivifying spent bone black by impregnating with hot solution of gelatin, drying and charring. **509,589**, November 28, Watel, E., process for purifying fatty matters, remaining after essential oils are extracted. **508,469**, November 14, Cross, A. K., crayon for polished surfaces; composed of "ozokerite, coloring matter, glycerol, aga aga, and soapy materials." **508,777**, November 14, Stearns, T. C., toilet soap; made from soap stock with sufficient almond meal to neutralize free alkali. **509,049**, November 21, Schicht, J., soap; caustic alkali is heated to 135°C., fat is heated separately to 100°, and the heated fat is injected into the hot alkali in fine jets and agitated.

NEW BOOKS.

A SELECT BIBLIOGRAPHY OF CHEMISTRY, 1492-1892. BY HENRY CARRINGTON BOLTON. (Smithsonian Miscellaneous Collections, Vol. 36.) Octavo, pp. XIII, 1212.

It is not too much to say of this book that one of greater usefulness to the chemist has not appeared within the last twenty-five years. The titles are given under the following heads: I, Bibliography, pp. 1-37; II, Dictionaries and Tables, including Nomenclature, pp. 38-84; III, History of Chemistry, pp. 84-170; IV, Biography, including Bibliographies of Individuals, pp. 171-257; V, Chemistry, Pure and Applied, pp. 258-942; VI, Alchemy, pp. 943-1067; VII, Periodicals, pp. 1068-1164; Addenda and Subject-Index, pp. 1165-1212. Titles in twenty-five languages are catalogued. Of these the titles of German books are most numerous; then come English, French, Latin, Italian, Dutch, Russian, Spanish, and Swedish. The other languages

have less than 100 titles each. Several languages are poorly represented—Finnish, Gujerathi, Hebrew, Hindi, Rumanian, and Welsh have but one each. For most of us, however, this single title will be one too many. The total number of titles indexed is 12,031, of which 273 belong to I, 327 to II, 730 to III, 979 to IV, 8,206 to V, 1,046 to VI, and 470 to VII.

Of Section V, Chemistry, Pure and Applied, Dr. Bolton says: While it "is more extensive than the other six combined, it is the least satisfactory to the editor. Besides pure chemistry it comprises works in every department of chemistry applied to the arts, but not works on the arts themselves; thus agricultural chemistry, but not agriculture; pharmaceutical chemistry, physiological chemistry, etc., find place, but not the sciences of pharmacy and medicine. It is, of course, often impossible to draw the line sharply, and in doubtful cases the tendency has been to include rather than exclude; hence many works on technology are included especially when chemistry lies at their foundation. It is probable, however, that this has not been done uniformly, owing to the difficulty of selection.

"Readers should not expect to find in this section complete lists of the works of a given author; though some pains have been taken in the case of prominent men to catalogue their writings fully (see under Berzelius, Fresenius, Liebig, Johnston, Orfila, Stockhardt, etc). The names of many eminent chemists will be found wanting in Section V, simply because they have pulbished no independent works, although voluminous contributors to periodicals."

The place of publication has been given whenever it could be ascertained but in many cases, perhaps in the majority of cases, not the name of the publisher. In many cases, too, the number of pages is not given—probably it could not be ascertained.

The value of the book is greatly increased by an admirable subject-index. E. H.

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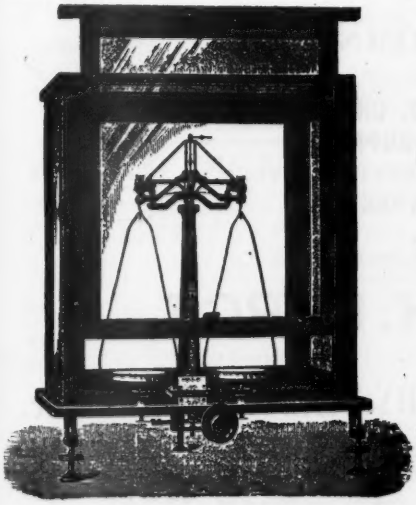

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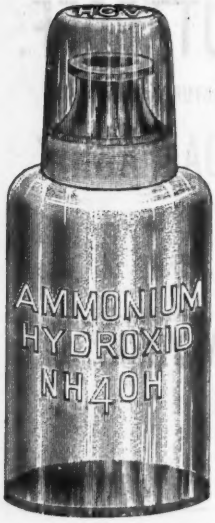

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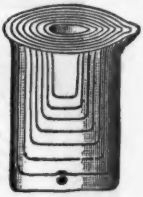

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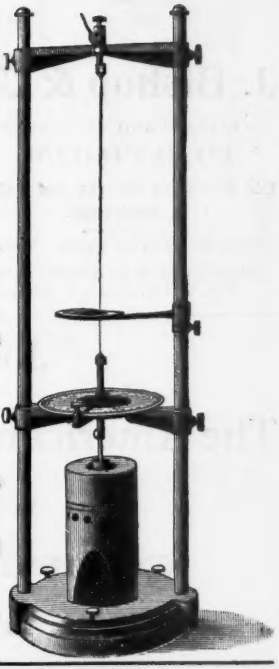

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